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PATENT ABSTRACTS OF JAPAN

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(72)Inventor : KANOYA IZURU
HOSOE MITSUYA
SUZUKI TAKANORI

0)Priority

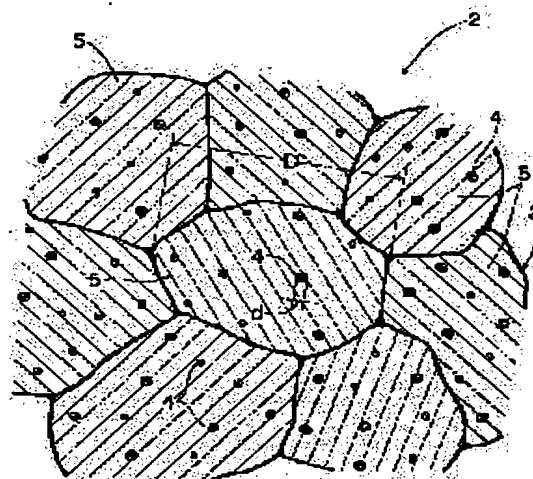
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4) HYDROGEN STORAGE ALLOY POWDER AND ON-VEHICLE HYDROGEN STORAGE TANK

7)Abstract:

ROBLEM TO BE SOLVED: To provide hydrogen storage alloy powder
ge in the quantity of hydrogen to be occluded, further high in hydrogen
cluding and discharging rate and suitable as the on-vehicle one.

OLUTION: Hydrogen storage alloy powder 1 is an aggregate of an Mg
atrix 3 and plural ultra-fine particles 4 dispersed into the Mg matrix 3.
ie Mg matrix 3 contains plural Mg crystals 5 having a particle diameter D
1.0 to 500 μ m. The particle diameter (d) of the ultra-fine particles is 10
500 nm. The ultra-fine particles 4 are composed of at least one kind
lected from among Ni ultra-fine particles, Fe ultra-fine particles, V
tra-fine particles, Mn ultra-fine particles, Ti ultras-fine particles, Cu
tra-fine particles, Al ultra-fine particles, Pd ultra-fine particles, Pt
tra-fine particles, Zr ultra-fine particles, Au ultra-fine particles, Ag
tra-fine particles, Co ultra-fine particles, Mo ultra-fine particles, Nb
tra-fine particles, Cu ultra-fine particles, Zn ultra-fine particles, Ru
tra-fine particles, Rh ultra-fine particles, Ta ultra-fine particles, Ir ultra-
ne particles, W ultra-fine particles and their alloy ultra-fine particles.



LEGAL STATUS

Date of request for examination]

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registration]

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AIMS

claim(s)]

claim 1] Are the aggregate of an alloy particle (2) which has two or more ultrafine particles (4) distributed to Mg matrix (3) and its Mg matrix (3), and the aforementioned Mg matrix (3) two or more Mg crystals (5) whose diameters of crystal grain are $1.0 \text{ micrometer} \leq D \leq 500 \text{ micrometer}$ It has and the particle size d of the aforementioned ultrafine particle (4) is $10 \text{ nm} \leq d \leq 500 \text{ nm}$. the aforementioned ultrafine particle (4) nickel ultrafine particle, nickel alloy ultrafine particle, Fe ultrafine particle, Fe alloy ultrafine particle, V ultrafine particle, V alloy ultrafine particle, Ti ultrafine particle, Mn alloy ultrafine particle, Ti ultrafine particle, Ti alloy ultrafine particle, Cu ultrafine particle, Cu alloy ultrafine particle, aluminum ultrafine particle, aluminum alloy ultrafine particle, Pd ultrafine particle, Pd alloy ultrafine particle, Pt ultrafine particle, Pt alloy ultrafine particle, Zr ultrafine particle, Zr alloy ultrafine particle, Au ultrafine particle, Au alloy ultrafine particle, Ag ultrafine particle, Ag alloy ultrafine particle, Co ultrafine particle, Co alloy ultrafine particle, Mo ultrafine particle, Mo alloy ultrafine particle, Nb ultrafine particle, Nb alloy ultrafine particle, Cr ultrafine particle, Cr alloy ultrafine particle, Zn ultrafine particle, Zn alloy ultrafine particle, Ru ultrafine particle, Ru alloy ultrafine particle, Rh ultrafine particle, Rh alloy ultrafine particle, Ta ultrafine particle, Ta alloy ultrafine particle, Ir ultrafine particle, Ir alloy ultrafine particle, W ultrafine particle And hydrogen storing metal alloy powder characterized by the thing which is chosen from W alloy ultrafine particle, and which is a kind at least.

claim 2] Hydrogen storing metal alloy powder according to claim 1 whose particle size d of the aforementioned ultrafine particle (4) is $d \geq 100 \text{ nm}$.

claim 3] Content GP of the aforementioned ultrafine particle (4) Hydrogen storing metal alloy powder according to claim 1 or 2 which is $0.1 \text{ atom \%} \leq GP \leq 5.0 \text{ atom \%}$.

claim 4] Content GP of the aforementioned ultrafine particle (4) Hydrogen storing metal alloy powder according to claim 1 or 2 which is $0.3 \text{ atom \%} \leq GP \leq 3.0 \text{ atom \%}$.

claim 5] Two or more aforementioned ultrafine particles (4) are hydrogen storing metal alloy powder according to claim 1, 2, 3, or 4 which consists of two or more nickel ultrafine particle and two or more Fe ultrafine particles.

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DETAILED DESCRIPTION

Detailed Description of the Invention]

[001]

In the technical field to which invention belongs] this invention relates to the hydrogen storage tank for mount having hydrogen storing metal alloy powder and its hydrogen storing metal alloy powder.

[002]

Description of the Prior Art] Conventionally, what is depended on mechanical alloying as hydrogen storing metal alloy powder, the thing pass each process of casting, trituration, and activation are known.

[003]

Problem(s) to be Solved by the Invention] However, the conventional hydrogen storing metal alloy powder had the problem of being inadequate, about hydrogen storage capacity and hydrogen-absorption discharge speed, in order to be used it as hydrogen storing metal alloy powder for mount in fuel cell loading vehicles.

[004]

Means for Solving the Problem] While hydrogen storage capacity is size, hydrogen-absorption discharge speed is quick, and this invention aims at offering the aforementioned hydrogen storing metal alloy powder suitable as an object for mount.

[005] In order to attain the aforementioned purpose According to this invention, Mg matrix And two or more ultrafine particles distributed to the Mg matrix It is the aggregate of the alloy particle which it has, and the aforementioned Mg matrix has two or more Mg crystals whose diameters D of crystal grain are $1.0 \text{ micrometer} \leq D \leq 500 \text{ micrometer}$, and the particle size d of the aforementioned ultrafine particle is $10 \text{ nm} \leq d \leq 500 \text{ nm}$. the aforementioned ultrafine particle is nickel ultrafine particle, nickel alloy ultrafine particle, Fe ultrafine particle, Fe alloy ultrafine particle, V ultrafine particle, V alloy ultrafine particle, Mn ultrafine particle, Mn alloy ultrafine particle, Ti ultrafine particle, Ti alloy ultrafine particle, Cu ultrafine particle, Cu alloy ultrafine particle, aluminum ultrafine particle, aluminum alloy ultrafine particle, Pd ultrafine particle, Pd alloy ultrafine particle, Pt ultrafine particle, Pt alloy ultrafine particle, Zr ultrafine particle, Zr alloy ultrafine particle, Au ultrafine particle, Au alloy ultrafine particle, Ag ultrafine particle, Ag alloy ultrafine particle, Co ultrafine particle, Co alloy ultrafine particle, Mo ultrafine particle, Mo alloy ultrafine particle, Nb ultrafine particle, Nb alloy ultrafine particle, Cr ultrafine particle, Cr alloy ultrafine particle, Zn ultrafine particle, The hydrogen storing metal alloy powder which is chosen from Zn alloy ultrafine particle, Ru ultrafine particle, Ru alloy ultrafine particle, Rh ultrafine particle, Rh alloy ultrafine particle, Ta ultrafine particle, Ta alloy ultrafine particle, Ir ultrafine particle, Ir alloy ultrafine particle, W ultrafine particle, and W alloy ultrafine particle and which is a kind at least is offered.

[006] As mentioned above, hydrogen storing metal alloy powder presents the hydrogen storage capacity as for which originates in the detailed metal texture, and has very high activity in Mg matrix which has the diameter D of crystal grain of nm order, therefore activation becomes it nothing size since particle size d is the aggregate of an alloy particle which distributed the ultrafine particle of nm order, and a quick hydrogen-absorption discharge speed.

[007] However, since the aforementioned diameter D of crystal grain becomes complicated [the manufacture process of hydrogen storing metal alloy powder] in $D < 1.0 \text{ micrometers}$, mass production becomes difficult, and on the other hand by $D > 500 \text{ micrometers}$, the hydrogen-absorption discharge speed of hydrogen storing metal alloy powder falls. Moreover, in $d < 10 \text{ nm}$, the activity of an ultrafine particle has the aforementioned, too high particle size d, the handling of the particle becomes difficult, and, on the other hand, the hydrogen storage capacity of hydrogen storing metal alloy powder serves as smallness by $d > 500 \text{ nm}$, and hydrogen-absorption discharge speed becomes slow.

[008] According to this invention, moreover, a Ti-Fe alloy matrix And it is the aggregate of an alloy particle which has two or more ultrafine particles distributed to the Ti-Fe alloy matrix. The aforementioned Ti-Fe alloy matrix has two or more Ti-Fe alloy crystals whose diameters D of crystal grain are $1.0 \text{ micrometer} \leq D \leq 500 \text{ micrometer}$, and the

particle size d of the aforementioned ultrafine particle is $10\text{ nm} \leq d \leq 500\text{ nm}$. the aforementioned ultrafine particle (4) includes ultrafine particle, nickel alloy ultrafine particle, Fe ultrafine particle, Fe alloy ultrafine particle, V ultrafine particle, V alloy ultrafine particle, Mn ultrafine particle, Mn alloy ultrafine particle, Ti ultrafine particle, Ti alloy ultrafine particle, Cu ultrafine particle, Cu alloy ultrafine particle, aluminum ultrafine particle, aluminum alloy ultrafine particle, Pd ultrafine particle, Pd alloy ultrafine particle, Pt ultrafine particle, Pt alloy ultrafine particle, Zr ultrafine particle, Zr alloy ultrafine particle, Au ultrafine particle, Au alloy ultrafine particle, Ag ultrafine particle, Ag alloy ultrafine particle, Co ultrafine particle, Co alloy ultrafine particle, Mo ultrafine particle, Mo alloy ultrafine particle, Nb ultrafine particle, Nb alloy ultrafine particle, Cr ultrafine particle, Cr alloy ultrafine particle, Zn ultrafine particle, The hydrogen storing metal alloy powder which is chosen from Zn alloy ultrafine particle, Ru ultrafine particle, Ru alloy ultrafine particle, Rh ultrafine particle, Rh alloy ultrafine particle, Ta ultrafine particle, Ta alloy ultrafine particle, Ir ultrafine particle, Ir alloy ultrafine particle, W ultrafine particle, and W alloy ultrafine particle and which is a kind at least is offered.

[009] This hydrogen storing metal alloy powder as well as the above presents without activation the hydrogen storage capacity which becomes size, and a quick hydrogen-absorption discharge speed. The reason for limitation of the aforementioned diameter D of crystal grain and the aforementioned particle size d is the same as the aforementioned case.

[010] Furthermore, according to this invention, it is a hydrogen storage tank for mount having hydrogen storing metal alloy powder. the aforementioned hydrogen storing metal alloy powder Mg matrix And two or more ultrafine particles distributed to the Mg matrix It is the aggregate of the alloy particle which it has, and the aforementioned Mg matrix has two or more Mg crystals whose diameters D of crystal grain are $1.0\text{ micrometer} \leq D \leq 500\text{ micrometer}$, and the particle size d of the aforementioned ultrafine particle is $10\text{ nm} \leq d \leq 500\text{ nm}$. the aforementioned ultrafine particle includes ultrafine particle, nickel alloy ultrafine particle, Fe ultrafine particle, Fe alloy ultrafine particle, V ultrafine particle, V alloy ultrafine particle, Mn ultrafine particle, Mn alloy ultrafine particle, Ti ultrafine particle, Ti alloy ultrafine particle, Cu ultrafine particle, Cu alloy ultrafine particle, aluminum ultrafine particle, aluminum alloy ultrafine particle, Pd ultrafine particle, Pd alloy ultrafine particle, Pt ultrafine particle, Pt alloy ultrafine particle, Zr ultrafine particle, Zr alloy ultrafine particle, Au ultrafine particle, Au alloy ultrafine particle, Ag ultrafine particle, Ag alloy ultrafine particle, Co ultrafine particle, Co alloy ultrafine particle, Mo ultrafine particle, Mo alloy ultrafine particle, Nb ultrafine particle, Nb alloy ultrafine particle, Cr ultrafine particle, Cr alloy ultrafine particle, Zn ultrafine particle, Zn alloy ultrafine particle, Ru ultrafine particle, Ru alloy ultrafine particle, The hydrogen storage tank for mount which is chosen from Rh ultrafine particle, Rh alloy ultrafine particle, Ta ultrafine particle, Ta alloy ultrafine particle, Ir ultrafine particle, Ir alloy ultrafine particle, W ultrafine particle, and W alloy ultrafine particle and which is a kind at least is offered.

[011] Since this hydrogen storage tank contains the hydrogen storing metal alloy powder which presents the hydrogen storage capacity which becomes size as mentioned above, and a quick hydrogen-absorption discharge speed, it is suitable as an object for mount. The reason for limitation of the aforementioned diameter D of crystal grain and the aforementioned particle size d is the same as the aforementioned case.

[012]

[Embodiments of the Invention] In drawing 1 and 2, the hydrogen storing metal alloy powder 1 is the aggregate of the alloy particle 2, and the alloy particle 2 has two or more ultrafine particles 4 distributed to the Mg matrix 3 and its Mg matrix 3. two or more Mg crystals 5 whose diameters D of crystal grain of the Mg matrix 3 are $1.0\text{ micrometer} \leq D \leq 500\text{ micrometer}$ -- having -- moreover, the particle size d of an ultrafine particle 4 -- $10\text{ nm} \leq d \leq 500\text{ nm}$ is $\leq 100\text{ nm}$ preferably In this case, let the diameter D of crystal grain of the Mg crystal 5, and particle size d of an ultrafine particle 4 be the length of those longest portions in a microphotography. This is below the same. Although only Mg crystals 5 with the aforementioned diameter D of crystal grain are consisted of by the Mg matrix 3, what has one which does not have the aforementioned diameter D of crystal grain outside, or two Mg crystals or more is contained in it. In the Mg matrix 3, about 50 percent of Mg crystals 5 with the aforementioned diameter D of crystal grain should just exist.

[013] As an ultrafine particle 4 **, nickel ultrafine particle, nickel alloy ultrafine particle, Fe ultrafine particle, Fe alloy ultrafine particle, V ultrafine particle, V alloy ultrafine particle, Mn ultrafine particle, Mn alloy ultrafine particle, Ti ultrafine particle, Ti alloy ultrafine particle, Cu ultrafine particle, Cu alloy ultrafine particle, aluminum ultrafine particle, aluminum alloy ultrafine particle, Pd ultrafine particle, Pd alloy ultrafine particle, Pt ultrafine particle, Pt alloy ultrafine particle, Zr ultrafine particle, Zr alloy ultrafine particle, Au ultrafine particle, Au alloy ultrafine particle, Ag ultrafine particle, Ag alloy ultrafine particle, Co ultrafine particle, Co alloy ultrafine particle, Mo ultrafine particle, Mo alloy ultrafine particle, Nb ultrafine particle, Nb alloy ultrafine particle, Cr ultrafine particle, Cr alloy ultrafine particle, Ru ultrafine particle, Zn alloy ultrafine particle, Ru ultrafine particle, Ru alloy ultrafine particle, Rh ultrafine particle,

alloy ultrafine particle, Ta ultrafine particle, Ta alloy ultrafine particle, Ir ultrafine particle, Ir alloy ultrafine particle, W ultrafine particle And as being chosen from W alloy ultrafine particle, a kind is used as it is few.

014] As mentioned above, the hydrogen storing metal alloy powder 1 presents the hydrogen storage capacity which originates in the detailed metal texture, and has very high activity in the Mg matrix 3 which has the diameter D of crystal grain of nm order, therefore becomes its size since particle size d is the aggregate of the alloy particle 2 which distributed the ultrafine particle 4 of nm order, and a quick hydrogen-absorption discharge speed.

015] It is the content GP of an ultrafine particle 4 that more than hydrogen-storage-capacity 6wt% should be secured. is set as 0.1 atom % \leq GP \leq 5.0 atom %. This content GP The meaning which uses an ultrafine particle 4 in GP < 0.1 atom % is lost, and, on the other hand, hydrogen storage capacity becomes less than [6wt%] by GP > 5.0 atom %. content GP of an ultrafine particle 4 It is 0.3 atom % \leq GP \leq 3.0 atom % preferably, and, thereby, more than hydrogen-storage-capacity 7.0wt% can be secured.

016] As hydrogen storing metal alloy powder 1, the aggregate of the alloy particle 2 which is distributed to the Ti-Fe alloy matrix 3 and its Ti-Fe alloy matrix 3 and which has two or more of the same ultrafine particles 4 as the above can be mentioned out of the aforementioned thing. in this case, two or more Ti-Fe alloy crystals 5 whose diameters D of crystal grain of the Ti-Fe alloy matrix 3 are 1.0 micrometer \leq D \leq 500 micrometer -- having -- moreover, the particle size d of an ultrafine particle 3 -- 10nm \leq d \leq -- 500nm is d \geq 100nm preferably Moreover, content GP of an ultrafine particle 4 since it is the same as that of the aforementioned thing It is 0.1 atom % \leq GP \leq 5.0 atom %, and is 0.3 atom % \leq GP \leq 3.0 atom % preferably. Although it consists of only Ti-Fe alloy crystals 5 which have the aforementioned diameter D of crystal grain in the Ti-Fe alloy matrix 3, one which does not have the aforementioned diameter of crystal grain outside, or the thing which has two or more Ti-Fe alloy crystals is also contained. In the Ti-Fe alloy matrix 3, about 50 percent of Ti-Fe alloy crystals 5 with the aforementioned diameter D of crystal grain should just exist.

017] In manufacture of the hydrogen storing metal alloy powder 1, the method of performing either mechanical alloying or mechanical grinding using the ultrafine particle powder which is the aggregate of the Ti-Fe alloy matrix powder which is the aggregate of a Ti-Fe alloy matrix particle, and an ultrafine particle 4, using the ultrafine particle powder which is the aggregate of Mg matrix powder which is the aggregate of Mg matrix particle, and an ultrafine particle 4 is adopted. In this case, particle size D0 of Mg matrix particle and a Ti-Fe alloy matrix particle 0 \leq 2000 micrometers of 10 micrometer \leq D are suitable. Particle size D0 In D0 < 10 micrometer, since the activity of these particles is high, handling nature becomes bad, on the other hand by D0 > 2000 micrometer, the hydrogen storage capacity of hydrogen storing metal alloy powder serves as smallness, and hydrogen-absorption discharge speed comes slow.

018] Hereafter, an example is explained.

019] [Example 1] Purity is 99.9% and it is particle size D0. With the aggregate of Mg matrix particle which is D0 < 500 micrometer, purity is 99.9%, particle size d is [the aggregate of 20 nm \leq d \leq 400nm (200nm of mean particle diameters) nickel ultrafine particle and purity] 99.9%, and particle size d prepared the aggregate of 100 nm \leq d \leq 500nm (300nm of mean particle diameters) Fe ultrafine particle. And weighing capacity of these particles is carried out so that the sum of the content of the aggregate of nickel ultrafine particle and the aggregate of Fe ultrafine particle to the aggregate of Mg matrix particle might change within the limits of 0.05 atom % to 10.0 atom % and it might be set to the interior division injury of the sum of each content, and nickel ultrafine particle:Fe ultrafine particle \approx 2:1, and the mixed-powder end of 15 sorts which it is 100g in total, and differs in a compounding ratio was obtained.

020] Each end of mixed powder is put into a pot (product made from JIS SUS316) with a capacity [of a horizontal-type ball mill (product made from Honda)] of 2500ml with 990 balls (product made from JIS SUS316) with a diameter of 10mm, the inside of a pot is held in the hydrogen gas atmosphere of 1.0MPa(s), and it is a pot rotational frequency. 64rpm, milling time t Ball milling was performed on the conditions of 4 hours. In this case, in a pot, it is a rotational acceleration GP. Acceleration 0.2G 0.2 times as many as this had occurred. Hydrogen storing metal alloy powder was extracted in the atmosphere after ball milling.

021] Since it was hydrogenated in the ball milling process, each hydrogen storing metal alloy powder performed hydrogenation processing which performs vacuum length to them on 350 degrees C and the conditions of 1 hour, and, subsequently carried out the hydrogen-absorption examination which performs high-pressure hydrogen pressurization of 1.1MPa(s) from a vacua at the measurement temperature of 310 degrees C about each hydrogen storing metal alloy powder.

022] Table 1 is the sum GP of the content of the diameter D of crystal grain of Mg crystal in Mg matrix about samples 1-15 of each hydrogen storing metal alloy powder, nickel ultrafine particle, and Fe ultrafine particle (nickel+Fe and the remainder are Mg), i.e., the content of an ultrafine particle. It is the content GP to a row. An inner vision (nickel, Fe) and hydrogen storage capacity SH It is shown.

23]
ble 1]

素吸 合金 末	MgマトリックスにおけるMg結晶の結晶粒径D (μm)	Ni, Fe超微粒子の含有量G _P (原子%)			水素吸蔵量S _H (wt%)
		Ni+Fe	Ni	Fe	
例1	500 ≦ ≦ 800	0.05	0.033	0.017	5.4
例2	200 ≦ ≦ 450	0.10	0.067	0.033	6.8
例3	50 ≦ ≦ 400	0.20	0.134	0.066	6.8
例4	5 ≦ ≦ 400	0.30	0.200	0.100	7.4
例5	1.5 ≦ ≦ 280	0.50	0.333	0.167	7.4
例6	3 ≦ ≦ 350	0.70	0.467	0.233	7.4
例7	2 ≦ ≦ 330	1.00	0.667	0.333	7.3
例8	1.1 ≦ ≦ 350	1.50	1.000	0.500	7.2
例9	4 ≦ ≦ 390	2.00	1.333	0.667	7.2
例10	3 ≦ ≦ 400	3.00	2.000	1.000	7.0
例11	3.5 ≦ ≦ 410	4.00	2.667	1.333	6.4
例12	2.5 ≦ ≦ 300	5.00	3.333	1.667	6.2
例13	1.5 ≦ ≦ 320	6.00	4.000	2.000	5.3
例14	1.8 ≦ ≦ 250	8.00	5.333	2.667	5.0
例15	2 ≦ ≦ 100	10.00	6.667	3.333	4.9

024] Drawing 3 is based on Table 1 and is the sum (nickel+Fe) GP of the content of nickel ultrafine particle and Fe ultrafine particle. Hydrogen storage capacity SH A relation is graph-ized. So that clearly from Table 1 and drawing 3 in the case of Examples 2-12 It is in within the limits whose diameter D of crystal grain of Mg crystal in Mg matrix is 1.0 micrometer ≦ D ≦ 500 micrometer. Moreover, the sum of the aforementioned content (nickel+Fe), that is, content GP of hydrogen storage capacity SH since it is set as 0.10 atom % ≦ GP ≦ 5.0 atom % it raises to SH ≧ 6wt% -- having -- ** -- especially -- like Examples 4-10 -- the amount GP of contents if it is set as 0.3 atom % ≦ GP ≦ 3.0 atom % -- hydrogen storage capacity SH It is possible to raise further, as it was called 7.0wt% ≦ SH ≦ 7.4wt%.

025] Next, PCT measurement was performed about Examples 5 and 10 after the aforementioned dehydrogenation processing. Drawing 4 is Example 5 and drawing 5 shows the PCT property (convergence time ; during 5 minutes 310 degrees C, occlusion discharge) of Example 10, respectively. Hydrogen storage capacity SH of drawing 4 and 5 to the Examples 5 and 10 As it was called 7.0wt% 7.4wt(s)%, respectively, it is size, and hydrogen-absorption discharge speed is very quick, and it turns out that the highest hydrogen-absorption burst size is presented in the convergence time for 5 minutes.

026] [Example 2] Purity is 99.9% and it is particle size D0. With the aggregate of the Ti50Fe50 alloy matrix particle (numerical unit is atomic %) which is D0 ≦ 300 micrometer, purity is 99.9% and particle size d prepared the aggregate of 100 nm ≦ d ≦ 500 nm (300 nm of mean particle diameters) Fe ultrafine particle. And content GP of the aggregate of Fe ultrafine particle to the aggregate of a Ti50Fe50 alloy matrix particle Weighing capacity was carried it so that it might become GP = 1 atom %, and the mixed-powder end of a total of 25g was obtained. This end of

ed powder is put into a pot (product made from JIS SUS316) with a capacity [of a horizontal-type ball mill
oduct made from Honda)] of 800ml with 180 balls (product made from JIS SUS316) with a diameter of 10mm, the
ide of a pot is held in the hydrogen gas atmosphere of 1.0MPa(s), and it is a pot rotational frequency. 85rpm, milling
e t Ball milling was performed on the conditions of 1 hour. In this case, in the pot, acceleration 0.1G 0.1 times as
ch as gravitational acceleration G had occurred. Hydrogen storing metal alloy powder was extracted in the
osphere after ball milling. The diameter D of crystal grain of the Ti50Fe50 alloy crystal in the Ti50Fe50 alloy
trix of this hydrogen storing metal alloy powder was $1.5 \text{ micrometer} \leq D \leq 450 \text{ micrometer}$. Let this end of an alloy
wder be Example 16.

027] Dehydrogenation processing was performed to Example 16 on the same conditions as an example 1,
sequently, Example 16 was held under the hydrogen pressure force of 1.1MPa(s) using PCT equipment, and change
the hydrogen pressure force accompanying time progress was measured. <: [A HREF="/Tokujitu/tjitemdrw.ipdl?
000=237&N0500=1E_N/;>;?] <6=9///&N0001=438&N0552=9&N0553=000009" TARGET="tjitemdrw"> drawing
hows a measurement result, and Example 17 is the same composition as Example 16, and is equivalent to the
ventional example by casting. Although the hydrogen pressure force is low in Example 16 so that clearly from
wing 6, it turns out that the occlusion of hydrogen is appearing simultaneously with hydrogen introduction. Since
hydrogen pressure force is low while Example 17 is subvital, the occlusion of hydrogen does not appear after 300-
ur progress.

028] [Example 3] Drawing 7 and hydrogen storage tank T for mount shown in 8 have the pressure-resistant outer
e object 6 of the cross-section round shape which consisted of stainless steel etc., and the inside of the outer case
ject 6 is divided with the permeability filter 7 made from nickel by the small space 9 by the side of the end wall 8,
d 11 between the skies by the side of the other end wall 10. The permeability filter 7 has the micropore of a large
mber which hydrogen can frequent, for example, the hole which is several nm - 0.5 micrometers. As a component of
permeability filter 7, metallic materials, such as stainless steel, ceramics, etc. are used out of nickel.

029] It fills up with the hydrogen storing metal alloy powder 1 in 11 between the skies, and the flow conduit 12 of
hydrogen for occlusion and discharge hydrogen penetrates, and is held at the end wall 8 by the side of the small
ace 9.

030] The shell 13 which consists of stainless steel etc. made the axis agree in the axis of the outer case object 6, and
s penetrated the outer case object 6, the permeability filter 7, and the hydrogen storing metal alloy powder 1, and the
al of the penetration section of the shell 13 in the ends walls 8 and 10 and the permeability filter 7 is carried out. The
side of a shell 13 is used as a path which circulates the fluid for cooling at the time of hydrogen absorption, and is
ed as a path which circulates a heating fluid at the time of hydrogen desorption.

031] The aforementioned powder is used as hydrogen storing metal alloy powder 1. That is, the powder is the
gregate of the alloy particle 2 which has two or more ultrafine particles 4 distributed to the Mg matrix 3 and its Mg
atrix 3. The Mg matrix 3 has two or more Mg crystals whose diameters D of crystal grain are $1.0 \text{ micrometer} \leq D$
500micrometer, and the particle size d of an ultrafine particle 4 is $10 \text{ nm} \leq d \leq 500 \text{ nm}$. Those ultrafine particles 4
kel ultrafine particle, nickel alloy ultrafine particle, Fe ultrafine particle, Fe alloy ultrafine particle, V ultrafine
rticle, V alloy ultrafine particle, Mn ultrafine particle, Mn alloy ultrafine particle, Ti ultrafine particle, Ti alloy
rafine particle, Cu ultrafine particle, Cu alloy ultrafine particle, aluminum ultrafine particle, aluminum alloy
rafine particle, Pd ultrafine particle, Pd alloy ultrafine particle, Pt ultrafine particle, Pt alloy ultrafine particle, Zr
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oy ultrafine particle, Co ultrafine particle, Co alloy ultrafine particle, Mo ultrafine particle, Mo alloy ultrafine
rticle, Nb ultrafine particle, Nb alloy ultrafine particle, Cr ultrafine particle, Cr alloy ultrafine particle, Zn ultrafine
rticle, Zn alloy ultrafine particle, Ru ultrafine particle, Ru alloy ultrafine particle, Rh ultrafine particle, Rh alloy
rafine particle, Ta ultrafine particle, Ta alloy ultrafine particle, Ir ultrafine particle, Ir alloy ultrafine particle, W
rafine particle And it is it a kind that it is few as being chosen from W alloy ultrafine particle.

032] Since this hydrogen storage tank T builds in the hydrogen storing metal alloy powder 1 which presents the
drogen storage capacity which becomes size as mentioned above, and a quick hydrogen-absorption discharge speed,
is suitable as an object for mount.

033]

ffect of the Invention] according to invention according to claim 1 to 9, hydrogen storage capacity is size by
nstituting as mentioned above -- both hydrogen-absorption discharge speed is quick, and hydrogen storing metal
oy powder suitable as an object for mount can be offered

034] According to invention according to claim 10, a hydrogen storage tank suitable as an object for mount can be
fered.

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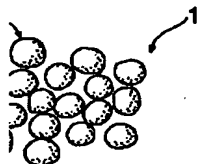
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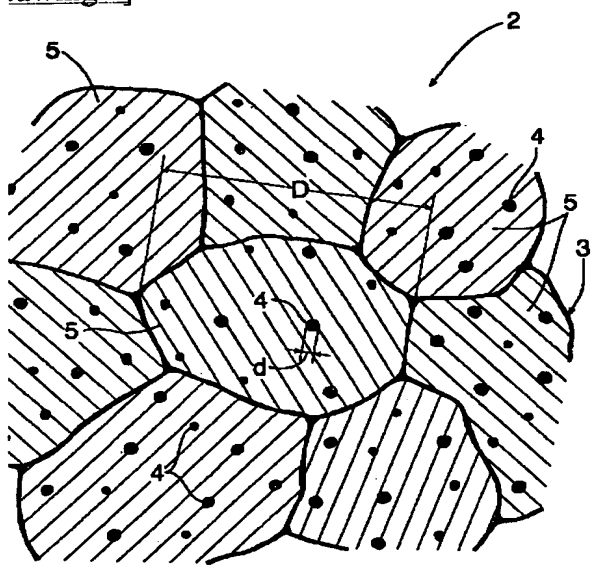
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DRAWINGS

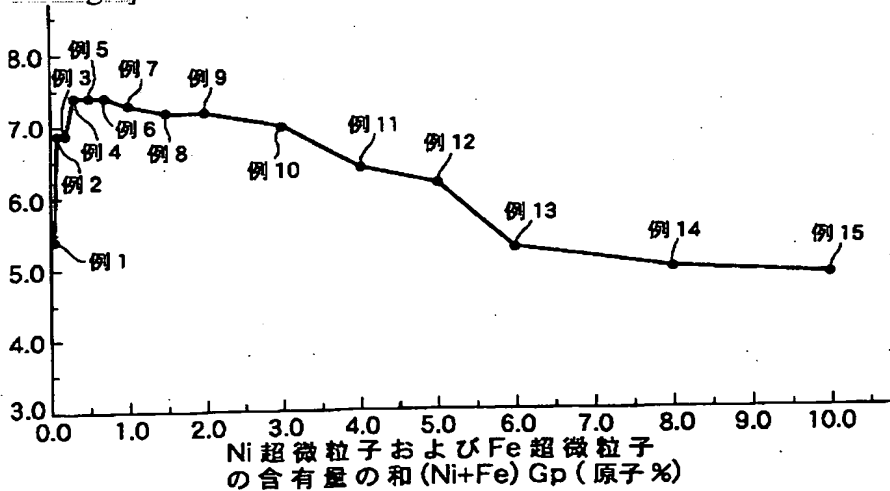
Drawing 1]



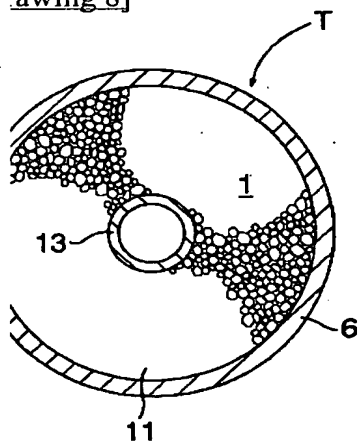
Drawing 2]



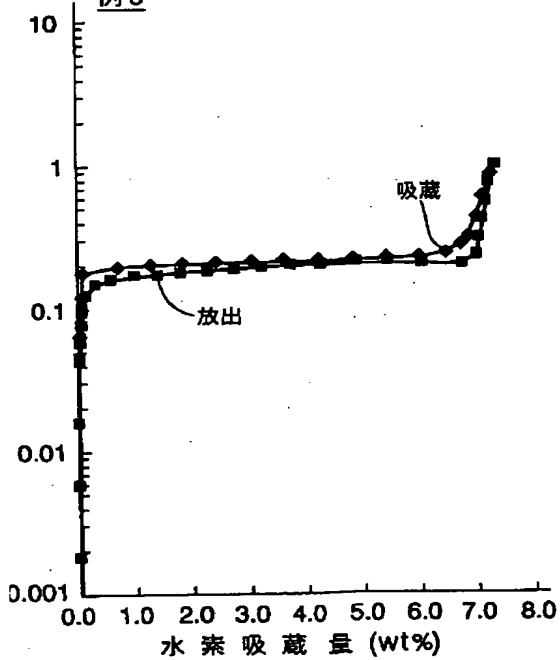
Drawing 3]



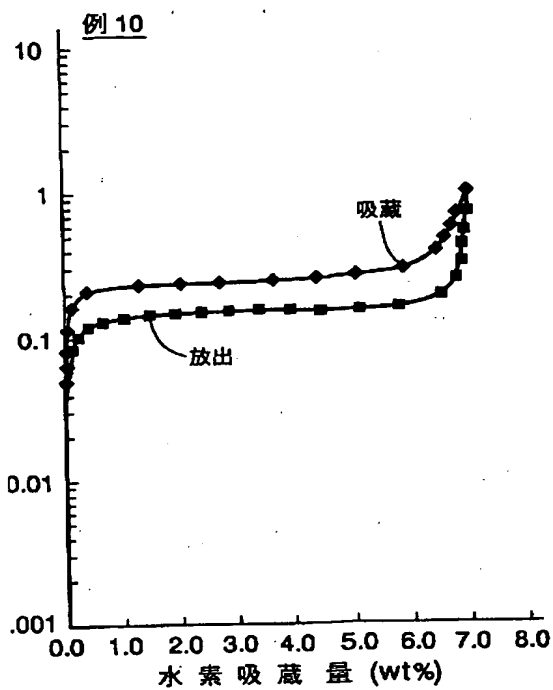
rawing 8]



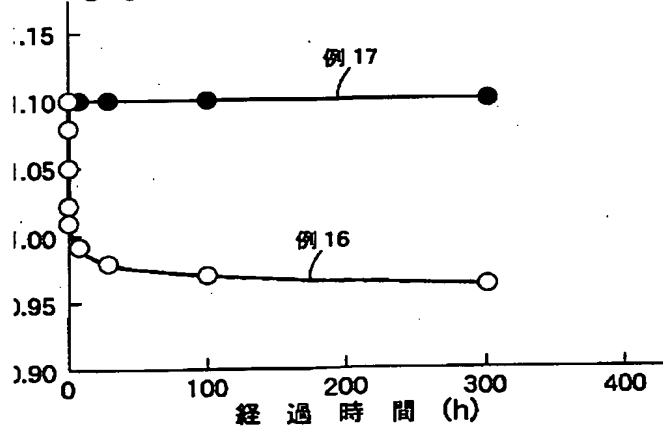
rawing 4]
例 5



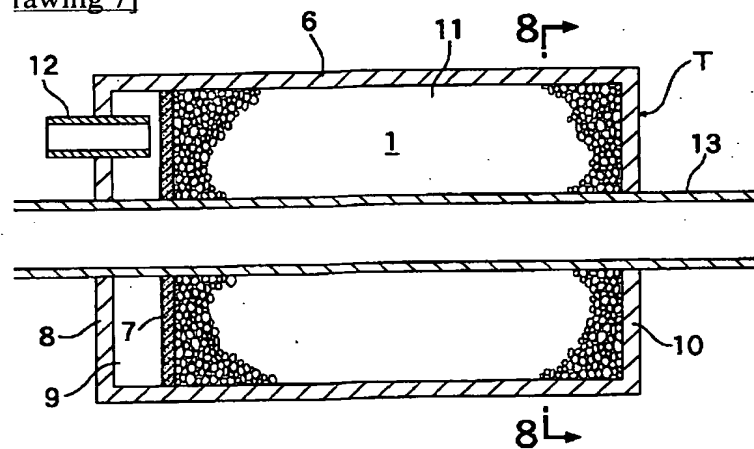
rawing 5]



rawing 6]



rawing 7]



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(33) 優先権主張国 日本 (J P)

(71) 出願人 000005326

本田技研工業株式会社

東京都港区南青山二丁目1番1号

(72) 発明者 鹿屋 出

埼玉県和光市中央1丁目4番1号 株式会

社本田技術研究所内

(72) 発明者 細江 光矢

埼玉県和光市中央1丁目4番1号 株式会

社本田技術研究所内

(74) 代理人 100071870

弁理士 落合 健 (外1名)

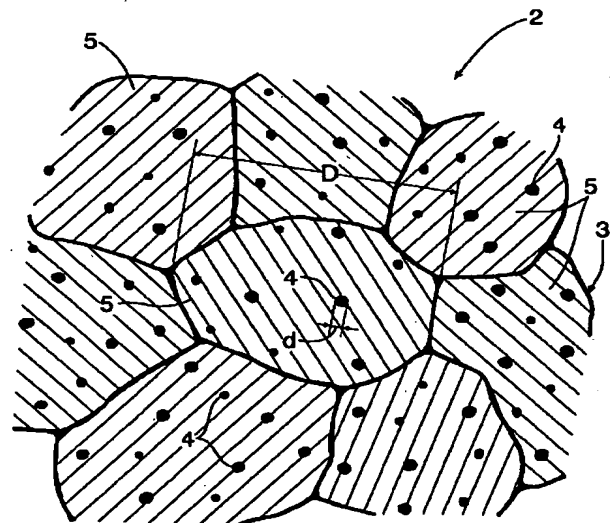
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(54) 【発明の名称】 水素吸蔵合金粉末および車載用水素貯蔵タンク

(57) 【要約】

【課題】 水素吸蔵量が大であると共に水素吸蔵放出速度が速く、車載用として好適な水素吸蔵合金粉末を提供する。

【解決手段】 水素吸蔵合金粉末1は、Mgマトリックス3およびそのMgマトリックス3に分散する複数の超微粒子4を有する合金粒子2の集合体である。Mgマトリックス3は結晶粒径Dが $1.0\mu\text{m} \leq D \leq 500\mu\text{m}$ である複数のMg結晶5を有する。超微粒子4の粒径dは $10\text{nm} \leq d \leq 500\text{nm}$ である。超微粒子4は、N i 超微粒子、F e 超微粒子、V 超微粒子、M n 超微粒子、T i 超微粒子、C u 超微粒子、A l 超微粒子、P d 超微粒子、P t 超微粒子、Z r 超微粒子、A u 超微粒子、A g 超微粒子、C o 超微粒子、M o 超微粒子、N b 超微粒子、C r 超微粒子、Z n 超微粒子、R u 超微粒子、R h 超微粒子、T a 超微粒子、I r 超微粒子、W 超微粒子およびそれらの合金超微粒子から選択される少なくとも一種である。



水素吸蔵合金粉末は、それを燃料電池搭載車両における車載用水素吸蔵合金粉末として用いるには、水素吸蔵量および水素吸蔵放出速度に関し不十分である、という問題があった。

【0004】

【課題を解決するための手段】本発明は、水素吸蔵量が大きく共に水素吸蔵放出速度が速く、車載用として好適な前記水素吸蔵合金粉末を提供することを目的とする。

【0005】前記目的を達成するため本発明によれば、MgマトリックスおよびそのMgマトリックスに分散する複数の超微粒子を有する合金粒子の集合体であって、前記Mgマトリックスは結晶粒径Dが $1.0\mu\text{m} \leq D \leq 500\mu\text{m}$ である複数のMg結晶を有し、また前記超微粒子の粒径dは $10\text{nm} \leq d \leq 500\text{nm}$ であり、前記超微粒子は、Ni超微粒子、Ni合金超微粒子、Fe超微粒子、Fe合金超微粒子、V超微粒子、V合金超微粒子、Mn超微粒子、Mn合金超微粒子、Ti超微粒子、Ti合金超微粒子、Cu超微粒子、Cu合金超微粒子、Al超微粒子、Al合金超微粒子、Pd超微粒子、Pd合金超微粒子、Pt超微粒子、Pt合金超微粒子、Zr超微粒子、Zr合金超微粒子、Au超微粒子、Au合金超微粒子、Ag超微粒子、Ag合金超微粒子、Co超微粒子、Co合金超微粒子、Mo超微粒子、Mo合金超微粒子、Nb超微粒子、Nb合金超微粒子、Cr超微粒子、Cr合金超微粒子、Zn超微粒子、Zn合金超微粒子、Ru超微粒子、Ru合金超微粒子、Rh超微粒子、Rh合金超微粒子、Ta超微粒子、Ta合金超微粒子、Ir超微粒子、Ir合金超微粒子、W超微粒子およびW合金超微粒子から選択される少なくとも一種である水素吸蔵合金粉末が提供される。

【0006】前記のように、水素吸蔵合金粉末は、 μm オーダーの結晶粒径Dを有するMgマトリックスに、粒径dがnmオーダーの超微粒子を分散させた合金粒子の集合体であることから、その微細金属組織に起因して非常に高い活性を有し、したがって、活性化処理無しに、大なる水素吸蔵量と速い水素吸蔵放出速度を呈する。

【0007】ただし、前記結晶粒径Dが $D < 1.0\mu\text{m}$ では水素吸蔵合金粉末の製造過程が複雑となるため量産が困難となり、一方、 $D > 500\mu\text{m}$ では水素吸蔵合金粉末の水素吸蔵放出速度が低下する。また前記粒径dが $d < 10\text{nm}$ では超微粒子の活性が高すぎて、その粒子の取扱いが難しくなり、一方、 $d > 500\text{nm}$ では水素吸蔵合金粉末の水素吸蔵量が小となり、また水素吸蔵放出速度が遅くなる。

【0008】また本発明によれば、Ti-Fe合金マトリックスおよびそのTi-Fe合金マトリックスに分散する複数の超微粒子を有する合金粒子の集合体であって、前記Ti-Fe合金マトリックスは結晶粒径Dが $1.0\mu\text{m} \leq D \leq 500\mu\text{m}$ である複数のTi-Fe合

金結晶を有し、また前記超微粒子の粒径dは $10\text{nm} \leq d \leq 500\text{nm}$ であり、前記超微粒子(4)は、Ni超微粒子、Ni合金超微粒子、Fe超微粒子、Fe合金超微粒子、V超微粒子、V合金超微粒子、Mn超微粒子、Mn合金超微粒子、Ti超微粒子、Ti合金超微粒子、Cu超微粒子、Cu合金超微粒子、Al超微粒子、Al合金超微粒子、Pd超微粒子、Pd合金超微粒子、Pt超微粒子、Pt合金超微粒子、Zr超微粒子、Zr合金超微粒子、Au超微粒子、Au合金超微粒子、Ag超微粒子、Ag合金超微粒子、Co超微粒子、Co合金超微粒子、Mo超微粒子、Mo合金超微粒子、Nb超微粒子、Nb合金超微粒子、Cr超微粒子、Cr合金超微粒子、Zn超微粒子、Zn合金超微粒子、Ru超微粒子、Ru合金超微粒子、Rh超微粒子、Rh合金超微粒子、Ta超微粒子、Ta合金超微粒子、Ir超微粒子、Ir合金超微粒子、W超微粒子およびW合金超微粒子から選択される少なくとも一種である水素吸蔵合金粉末が提供される。

【0009】この水素吸蔵合金粉末も前記同様に、活性化処理無しに、大なる水素吸蔵量と速い水素吸蔵放出速度を呈する。前記結晶粒径Dおよび前記粒径dの限定理由は前記の場合と同じである。

【0010】さらに本発明によれば、水素吸蔵合金粉末を内蔵した車載用水素貯蔵タンクであって、前記水素吸蔵合金粉末は、MgマトリックスおよびそのMgマトリックスに分散する複数の超微粒子を有する合金粒子の集合体であり、前記Mgマトリックスは結晶粒径Dが $1.0\mu\text{m} \leq D \leq 500\mu\text{m}$ である複数のMg結晶を有し、また前記超微粒子の粒径dは $10\text{nm} \leq d \leq 500\text{nm}$ であり、前記超微粒子は、Ni超微粒子、Ni合金超微粒子、Fe超微粒子、Fe合金超微粒子、V超微粒子、V合金超微粒子、Mn超微粒子、Mn合金超微粒子、Ti超微粒子、Ti合金超微粒子、Cu超微粒子、Cu合金超微粒子、Al超微粒子、Al合金超微粒子、Pd超微粒子、Pd合金超微粒子、Pt超微粒子、Pt合金超微粒子、Zr超微粒子、Zr合金超微粒子、Au超微粒子、Au合金超微粒子、Ag超微粒子、Ag合金超微粒子、Co超微粒子、Co合金超微粒子、Mo超微粒子、Mo合金超微粒子、Nb超微粒子、Nb合金超微粒子、Cr超微粒子、Cr合金超微粒子、Zn超微粒子、Zn合金超微粒子、Ru超微粒子、Ru合金超微粒子、Rh超微粒子、Rh合金超微粒子、Ta超微粒子、Ta合金超微粒子、Ir超微粒子、Ir合金超微粒子、W超微粒子およびW合金超微粒子から選択される少なくとも一種である車載用水素貯蔵タンクが提供される。

【0011】この水素貯蔵タンクは、前記のように大なる水素吸蔵量と速い水素吸蔵放出速度を呈する水素吸蔵合金粉末を内蔵しているので、車載用として好適である。前記結晶粒径Dおよび前記粒径dの限定理由は前記の場合と同じである。

【0021】各水素吸蔵合金粉末は、そのボールミリング過程において水素化されているので、それらに、350℃、1時間の条件で真空引きを行う脱水素化処理を施し、次いで、各水素吸蔵合金粉末について、測定温度310℃にて真空状態から1.1MPaの高圧水素加圧を行う水素吸蔵試験を実施した。

【0022】表1は、各水素吸蔵合金粉末の例1～15

に関するMgマトリックスにおけるMg結晶の結晶粒径D、Ni超微粒子およびFe超微粒子の含有量の和(Ni+Fe、残部はMg)、つまり超微粒子の含有量G_pならびにその含有量G_pの内分け(Ni、Fe)と、水素吸蔵量S_Hを示す。

【0023】

【表1】

水素吸蔵合金粉末	MgマトリックスにおけるMg結晶の結晶粒径D(μm)	Ni, Fe超微粒子の含有量G _p (原子%)			水素吸蔵量S _H (wt%)
		Ni+Fe	Ni	Fe	
例1	500 ≤ D ≤ 800	0.05	0.033	0.017	5.4
例2	200 ≤ D ≤ 450	0.10	0.067	0.033	6.8
例3	50 ≤ D ≤ 400	0.20	0.134	0.066	6.8
例4	5 ≤ D ≤ 400	0.30	0.200	0.100	7.4
例5	1.5 ≤ D ≤ 280	0.50	0.333	0.167	7.4
例6	3 ≤ D ≤ 350	0.70	0.467	0.233	7.4
例7	2 ≤ D ≤ 330	1.00	0.667	0.333	7.3
例8	1.1 ≤ D ≤ 350	1.50	1.000	0.500	7.2
例9	4 ≤ D ≤ 390	2.00	1.333	0.667	7.2
例10	3 ≤ D ≤ 400	3.00	2.000	1.000	7.0
例11	3.5 ≤ D ≤ 410	4.00	2.667	1.333	6.4
例12	2.5 ≤ D ≤ 300	5.00	3.333	1.667	6.2
例13	1.5 ≤ D ≤ 320	6.00	4.000	2.000	5.3
例14	1.9 ≤ D ≤ 250	8.00	5.333	2.667	5.0
例15	2 ≤ D ≤ 100	10.00	6.667	3.333	4.9

【0024】図3は表1に基づいてNi超微粒子およびFe超微粒子の含有量の和(Ni+Fe)G_pと水素吸蔵量S_Hとの関係をグラフ化したものである。表1、図3から明らかなように、例2～12の場合は、MgマトリックスにおけるMg結晶の結晶粒径Dが1.0μm ≤ D ≤ 500μmの範囲内にあり、また前記含有量の和(Ni+Fe)、つまり、含有量G_pが0.10原子% ≤ G_p ≤ 5.0原子%に設定されていることから、水素吸蔵量S_HはS_H ≥ 6wt%に高められており、特に、例4～10の如く、含量量G_pを0.3原子% ≤ G_p ≤ 3.0原子%に設定すると、水素吸蔵量S_Hを7.0wt%

t% ≤ S_H ≤ 7.4wt%といったように一層高めることが可能である。

【0025】次に、前記脱水素化処理後の例5、10についてPCT測定を行った。図4は例5の、また図5は例10のPCT特性(収束時間:5分間;310℃、吸蔵放出)をそれぞれ示す。図4、5から、例5、10の水素吸蔵量S_Hがそれぞれ7.4wt%、7.0wt%といったように大であり、また水素吸蔵放出速度が非常に速く、5分間の収束時間において最高水素吸蔵放出量を呈することが判る。

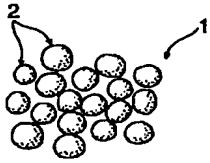
【0026】〔実施例2〕純度が99.9%であり、且

3.....Mgマトリックス, Ti-Fe合金マトリックス

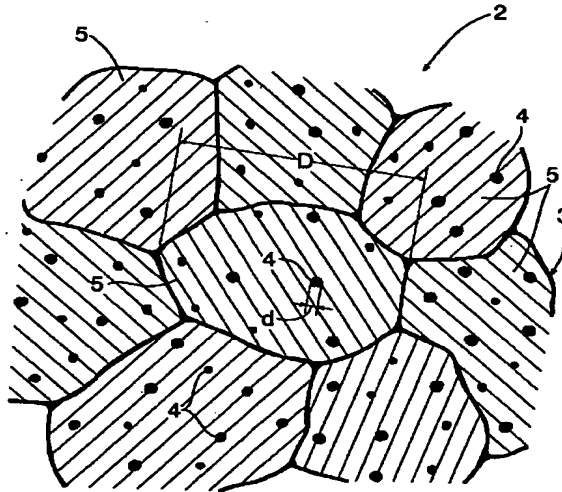
4.....超微粒子

T.....水素貯蔵タンク

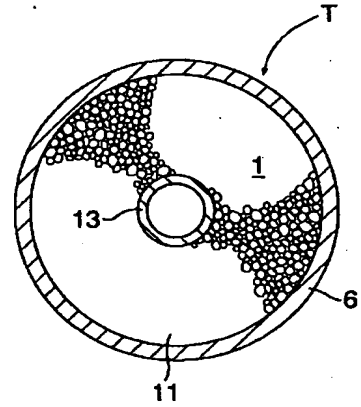
【図1】



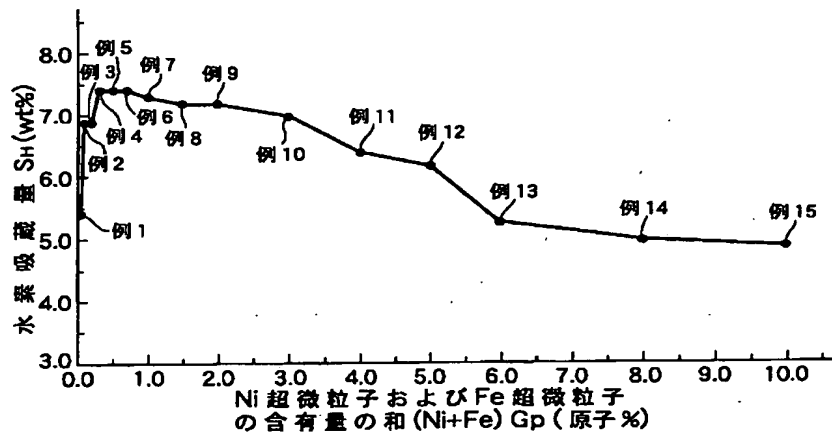
【図2】



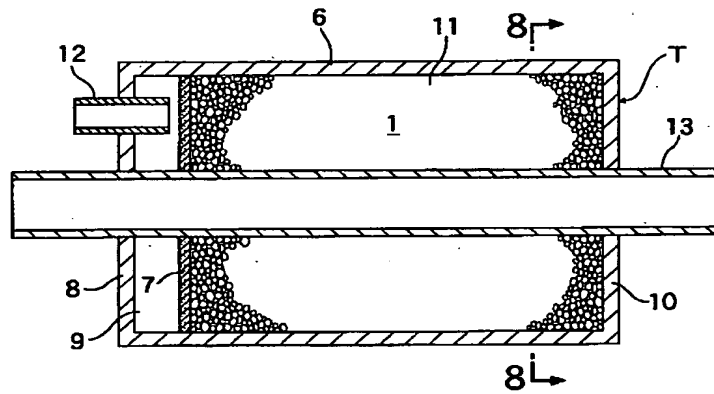
【図8】



【図3】



【図7】



フロントページの続き

(72) 発明者 鈴木 貴紀
埼玉県和光市中央1丁目4番1号 株式会
社本田技術研究所内

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